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The Hardening of Aluminous Cement at High and Low Temperatures.

By STEVEN GOTTLIEB.

As it is possible that in the course of the war it may be necessary, as has already happened in Finland, to construct concrete fortifications or other works at very low temperatures, the following account of concrete works successfully carried out at temperatures as low as 0 deg. F. may be useful. The results of tests on the hardening of aluminous cement at high temperatures are also given. Aluminous cement was also used for the construction work at low temperatures.

Effect of Heat on Mortar Cubes.

A typical analysis of the aluminous cement used was as follows :

	Per cent.
Loss on ignition	0·20
SiO_2	3·80
Al_2O_3, TiO_2	43·51
Fe_2O_3, FeO, Fe_3O_4	13·11
CaO	37·48
MgO	0·32
Insoluble	1·16
SO_3	0·40

The setting time of the cement at 65 deg. F. was : initial 5 hours, final 7 hours. In the tests 27 per cent. of water was used for normal consistency, corresponding to a standard penetration of 5 mm. to 7 mm. from the bottom of the Vicat mould. The tensile strength at 24 hours was 570 lb. per square inch, and the crushing strength at 24 hours was 9,500 lb. per square inch, on 1 : 3 mortar cubes gauged with 8 per cent. of water, and gauged and hardened at 65 deg. F.

In the first series of tests cubes with $2\frac{3}{4}$-in. sides were compacted with the Boehmé hammer. The cubes and moulds were immediately put into an insulated moist cupboard in which the temperature could be kept constant. One

hundred cubes were made for each test, and were crushed in groups of four after varying hardening periods, starting at 5½ hours and continuing at short intervals up to 48 hours. *Fig.* 1 shows the progress of hardening at temperatures of 21, 32, 65, 86, and 104 deg. F. ; at the three last temperatures special care was taken to keep the cubes constantly moist.

The well-known peculiarities of aluminous cements are clearly shown by the results. The typical points of maximum strength correspond to the times when most of the heat generation of the cement has taken place. *Fig.* 2 shows the rate of hardening of the mortar and the heat development of neat cement. Heat was measured in a Dewar vacuum vessel, the insulation of which is not perfect, and this explains the downward trend of the curve after maximum was reached. The heat developed by the cement promotes hardening to a certain degree.

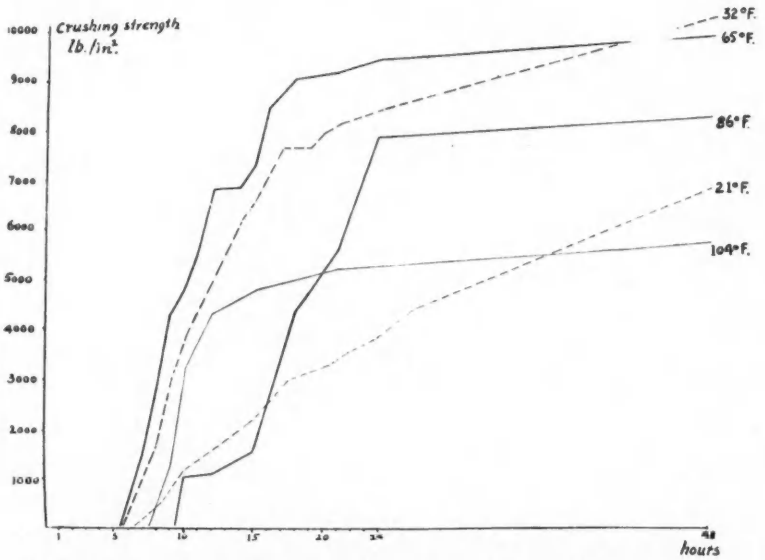

Fig. 1.—Rate of Hardening of 1 : 3 Aluminous Cement Mortar at Various Temperatures.

Fig. 3 shows the results of tests in which, after various times of hardening at 65 deg. F., 1 : 3 mortar cubes were heated up to 170 deg. F. for several hours in a moist atmosphere. In all cases the heating increased the initial strength, but the continued application of moist heat resulted in deterioration. At point No. 1 the cubes were put into the moist-heat cupboard after six hours' hardening ; at this early age the cement was very sensitive, resulting in high strength after ten hours, but there was a definite deterioration when the high temperature had acted for sixteen hours or more. With the progress of hardening the resistance

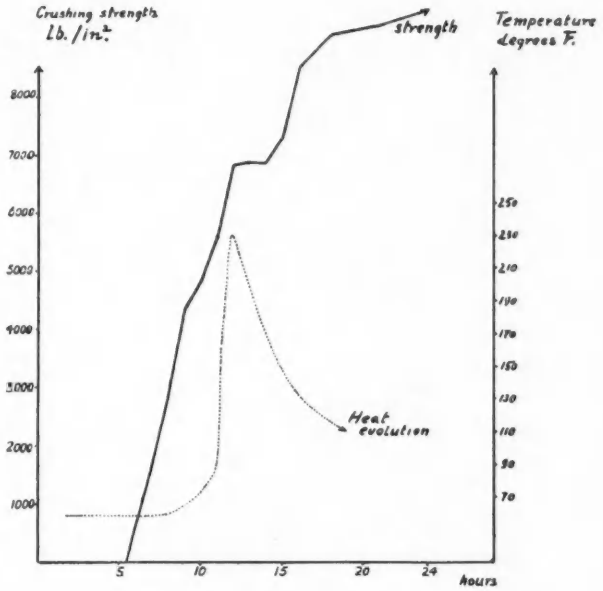

Fig. 2.—Rate of Hardening of 1 : 3 Aluminous Cement Mortar and Heat Evolution Curve of Neat Cement.

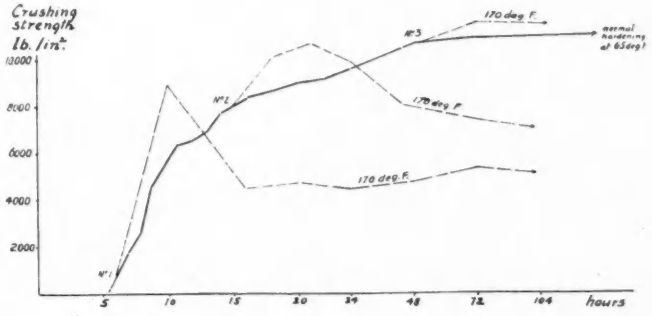

Fig. 3.—Aluminous Cement Mortar (1 : 3) Heated to 170 deg. F. after Different Hardening Periods at 65 deg. F.

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of the cubes against heat increased, as is seen when the cubes were put into the high-temperature cupboard after they had hardened for 15 and 48 hours at 65 deg. F. (see points Nos. 2 and 3).

Fig. 1 shows that, when heat was applied immediately after the cubes were prepared, even the relatively low temperature of 86 deg. F. gave less favourable results in the first 48 hours than a temperature of 65 deg. F. With somewhat higher water content, corresponding to a plastic consistency, the resistance of mortar against moist heat improved slightly. Those interested in these peculiarities of aluminous cements are referred to the works of Freyssinet and Coyne,[1] Roscher-Lund,[2] Bolomey,[3] Rengade[4] and Bates.[5]

The behaviour of hardened aluminous cement concrete or mortar in dry heat is entirely different. In this case it has a remarkable resistance; in fact

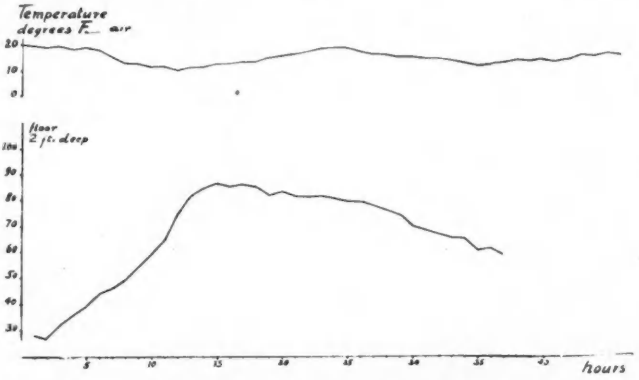

Fig. 4.—Temperature Changes during Construction.

aluminous cement concrete made with suitable aggregates is an excellent refractory up to temperatures of 2,300 deg. F.[6]

Hardening in Low Temperatures.

Aluminous cement concrete has exceptional hardening properties at temperatures below freezing point. It has been established in actual construction work on which the author was engaged, and where temperatures around 0 deg. F. continuously prevailed, that the temperature inside a floor 4ft. 6in. thick did not fall below 32 deg. F. at 4in. below its surface; at 2ft. below the surface the temperature did not fall below 40 deg. F. The shuttering was removed after 30 hours, and the concrete resounded well when struck with a hammer.

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[1] Freyssinet and Coyne: *Genie Civil*, 1927, No. 11.
[2] Roscher-Lund: *Zement*, 1927, No. 34; and 1928, Nos. 47 and 48.
[3] Bolomey: *Bulletin Technique*, 1927, No. 24.
[4] Rengade: Lecture at Congress on Testing Mat. Zürich, 1931. *Rev. des Mat. de Cons.*, 1927, p. 165.
[5] Bates: Lecture at Congress on Testing Mat. Zürich, 1931.
[6] Hussey: Refractory Concrete (Bulletin); *Cem. and Lime Manuf.*, 1937, p. 95.

On another contract 8-in. test tubes were cut out of a hardened wall and floor and tested. On this work air temperatures ranged between 10 deg. F. and 20 deg. F. (*Fig.* 4). The crushing strengths were as follows:

Cubes taken from wall 2ft. 7½in. thick: Inside, 6,560 lb. per sq. in.; outside, 3,550 lb. per sq. in.

Cubes taken from floor 4ft. 6in. thick: Upper part, 2,560 lb. per sq. in.; lower part, 5,340 lb. per sq. in.

Cubes taken from wall 11in. thick: 2,830 lb. per sq. in.

All the cubes were tested 40 hours after the concrete was placed. The concrete contained 640 lb. of cement per cu. yd., the maximum size of the aggregate was 1in., and the water-cement ratio was 0·60. When the upper parts of the floor were cast the concrete was mixed in a specially-designed heated mixer for at least 12 minutes, and the temperature of the mix was 35 to 40 deg. F.

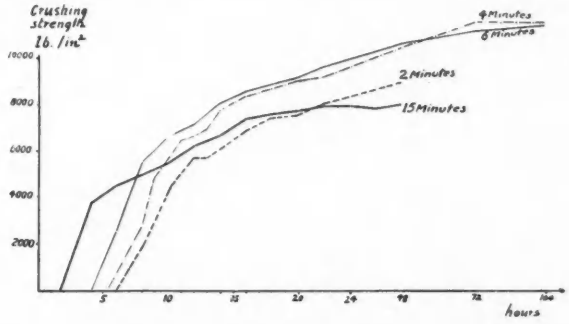


Fig. 5.—Influence of Various Mixing Times on Hardening.

By prolonged mixing a considerable shortening of the setting time was obtained, hardening starting 1¾ hours after casting. In spite of the temperature being continuously well below freezing point, all the surfaces were in excellent condition, and there was no sign of the action of frost.

When casting concrete in freezing weather the aggregates must never be used in a frozen condition, and the mix must be free from ice. Prolongation of mixing time has many advantages; even if the temperature of the concrete mix falls well below the freezing point of water, prolonged mixing improves the quality of the concrete, shortens the setting time, and expedites the development of heat and strength.

Fig. 5 shows the results of laboratory tests on 1 : 3 standard mixes of plastic consistency (with the addition of 11 per cent. of water). The mortars were mixed in a laboratory mixing machine for periods varying between two and fifteen minutes at air temperatures of 65 deg. F. The best results were obtained with mixing periods of four and six minutes; although mixing for fifteen minutes resulted in lower strength, hardening began earlier.

At temperatures of about 30 deg. F. tests were carried out with concrete containing 500 lb. of cement per cu. yd. and 1-in. aggregate, and having a water-cement ratio of 0·70. The concrete was mixed for 2, 4, and 15 minutes, and six cubes with 8-in. sides were cast from each mix. The results were as follows:

No.	Mixing time (minutes)	Crushing strength (lb. per sq. in.)		
		24 hours	48 hours	7 days
1	2	0	1,190	4,720
2	4	2,060	4,680	5,460
3	15	3,420	3,500	4,000

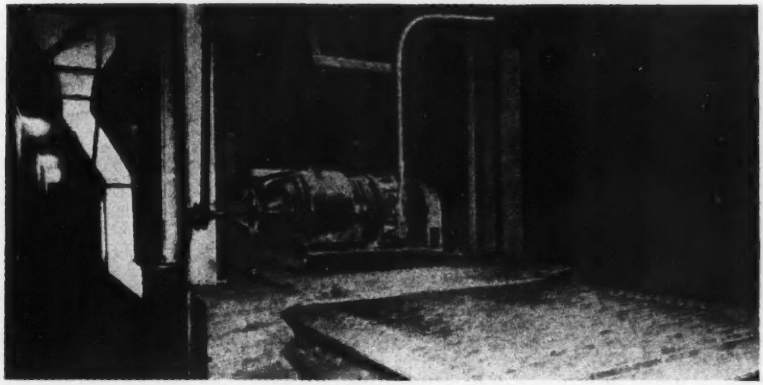
During the hardening of these cubes the temperature varied from 17 deg. F. to 22 deg. F. in the first 48 hours and from 17 deg. F. to 34 deg. F. between 48 hours and 7 days. After 48 hours the surfaces of Nos. 1 and 2 were frozen and thin layers up to ⅛in. did not harden at all. The surface of No. 3 hardened faultlessly.

Inclined-Grate Clinker Coolers.

SOME information on the performance of inclined-grate coolers at the Keystone Portland Cement Co.'s works at Bath, Penn., is given in a recent number of *Rock Products*. The works is operated on the wet process and has four 10ft. by 13ft. by 12ft. by 250ft. Polysius Solo kilns, two of which are now equipped with Fuller air-quenching coolers. The cooler consists of reciprocating

Kiln Hood rebuilt with Installation of New Clinker Cooler. Air Intake Fan on Left. Duct Leads to Inclined Grate.

and stationary grates which cause the clinker to assume a uniformly thin inclined bed in the direction of the discharge end. All but the driving mechanism is housed in a brick chamber below the kiln. The clinker from the kiln drops through a vertical chute and is distributed upon the grates and moved at a uniform rate to the low end. Fixed grates alternate with the reciprocating grates and both types are water-cooled by tubes around which each grate casting is poured. A single-drive shaft is driven through a speed-reducer by a variable-speed motor, the speed controlling the depth of the clinker and its rate of movement through the cooler. Cooling air enters through an air chamber and is forced through the clinker bed, both through spaces between the grates and through flared openings in each grate, thus subjecting the clinker particles to horizontal and vertical air blasts as they descend from grate to grate. The object is to provide long paths of air travel in contact with clinker particles for a maximum heat transfer with relation to the volume of air.



Drive End of New Clinker Cooler.

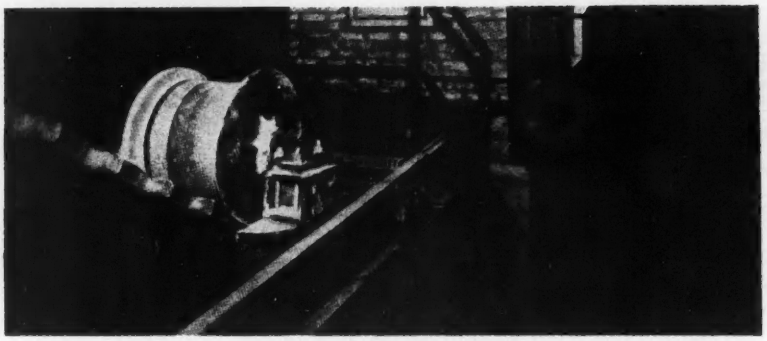
The coolers are of the combined recuperator and final cooling type with a divided chamber, and are placed at right angles to the kilns in a tunnel of very limited area. Each unit is 20ft. long, and the kilns are at 26ft. centres. Each grate has a surface area of 80 sq. ft. and operates with a steady power input of ¾ h.p. to 1 h.p. This cooling surface is designed for a kiln output of 1,750 barrels a day. Originally each 250ft. kiln had an enlarged burning zone 35ft. in length followed by a 26ft. length of cooling zone, which was very satisfactory as a heat recuperator and produced a clinker of about 675 deg. F. The burner pipe was 27 ft. long and air-cooled, with a 3ft. tip of heat-resistant metal.

The firing zone has been converted into an extension to the calcining zone, and is lined with 40 per cent. alumina brick, and the cooling zone is now the firing zone lined with 50 per cent. alumina brick. Combustion has been accelerated and burning is completed very close to the point of discharge of the clinker to

prevent delay in cooling and to allow a sudden drop in clinker temperature The burner pipe, which extends 3ft. inside the kiln hood, is water-cooled, and firing is done with a much shorter and hotter flame.

Air for quenching is forced through the hottest clinker at the upper end of the cooler, and the preheated air enters the kiln around the burner pipe as the secondary air for combustion. Additional air is forced through the secondary portion of the grate to reduce the final clinker temperature to about 350 deg. F. as compared with 675 deg. F. from the cooler which has been displaced. This is a sufficient reduction for efficient grinding since the clinker is rehandled and stored sufficiently for its temperature to drop between 100 deg. F. and 200 deg. F. before grinding. The temperature of the clinker averages about 2,400 deg. F. when it falls on the grate.

Other test data were compiled at the time these temperature readings were taken. The clinker bed averaged about 8in. to 9in. in depth and the stack gas



Discharge End of Cooler.

analysis was about the same as before the new coolers were installed. Exit stack temperatures were 650 to 700 deg. F., which also remained about constant. Primary air, taken from the tunnel below the kilns, entered the kiln with the pulverised coal at about 89 deg. F. and at the rate of about 4,000 cubic feet a minute, while about 16,300 cubic feet a minute passed through the cooler-quencher and was preheated to about 970 deg. F. before entering the kiln. About 9,300 cubic feet of air per minute was passed through the secondary part of the cooler and exhausted to the atmosphere at about 350 deg. F. This represents about 61,000 B.T.U. per minute, or about 4½ lb. of coal of the grade used.

A decrease in coal consumption has been observed, although accurate figures are not yet available. Autoclave expansions are about 0·25 per cent., using the standard mix of feed which had to be altered before in order to keep expansions down. The effect has been to allow the use of materials in the quarry which formerly were abandoned.

Magnesite in Kiln Linings

In the course of a lecture entitled "Magnesite," delivered by Mr. A. W. Comber, F.I.C., before the Institute of Chemistry of Great Britain and Ireland, as the Streatfeild Memorial Lecture, 1939, the author said two physical factors which have to be taken into account in firing magnesite to the refractory stage are melting point and specific gravity. There is a wide divergence in the published figures relating to the melting point of magnesia, either as the pure chemically-prepared compound or as the commercial material produced by calcination of some form of mineral magnesite. Moissan fused magnesia in the electric arc but his value of 3,000 deg. C. was an estimate and not a precise measurement. At the other extreme, Hempel gives the melting point of dead-burned Austrian breunnerite as 1,825–1,830 deg. C. There are many other figures recorded between these limits, and occasionally the differences are wide even when the same class of material was under examination.

Magnesia, whether pure or containing an appreciable amount of impurity, such as iron, when heated to a sufficiently high temperature after decomposition passes through a phase of progressively increasing plasticity, which continues over a range of some hundreds of degrees to the point of complete fusion. There appears to have been a lack of uniformity concerning the exact stage in this softening process, short of actual liquefaction, which should be considered as the melting point. For practical purposes the melting points of good quality dead-burned breunnerite and of pure magnesia may be taken as 2,000 deg. C. and 2,500 deg. C. respectively, but the figures are necessarily approximate.

The object in kiln practice is to produce, as closely as possible, an artificial form of periclase. This is a comparatively rare mineral form of magnesia, crystallising in cubes and octahedra. The generally accepted specific gravity of periclase is 3·67 but this figure is undoubtedly too high for pure crystalline magnesia. Actually it relates to a specimen containing 5·97 per cent. of ferrous oxide, and although, in a sense, this may bring it into rough correspondence with the dead-burned product from some grades of breunnerite, it is not altogether satisfactory as a basis for comparison. A more desirable standard is that given by J. H. Chesters, who determined the specific gravity of a crystal of fused pure magnesia, which was as transparent as glass. The value obtained was 3·583.

In considering the principles which govern the production of refractory magnesite, it will be an advantage first to consider briefly what happens when the amorphous mineral is calcined to a temperature high enough to cause sintering Amorphous magnesite of good quality contains 3 per cent. or less of impurities, which are chiefly calcium carbonate and silica. The metallic iron content may be below 0·2 per cent. In such case, the impurities do not greatly affect the temperature necessary to reach the stage of sintering or incipient fusion, which may be 1,800 deg. C. or even higher. Both chemical and physical changes are relatively simple. The special problems are those involved in reaching the temperature required and providing a suitably resistant kiln lining to withstand

it in operation. Further, when bricks are made from the effectively dead-burned product of a fairly pure amorphous magnesite, cohesion of the component particles appears due, to a large extent, to intercrystallisation or merging and not primarily to the cementing action of a bonding medium of different composition from the rest of the brick derived from impurities in the mineral.

When breunnerite is calcined other considerations become operative. The iron enters into combination as magnesioferrite, $MgO.Fe_2O_3$, which has many points of resemblance to magnetite, $FeO.Fe_2O_3$, in colour, crystalline structure, and in their common magnetic properties. The two compounds may have an isomorphous relationship, although evidence of the existence of intermediates, with MgO and FeO as variables, is not very positive. The effect of the formation of magnesioferrite is to reduce the sintering temperature, which may be as low as 1,450 deg. C., but is more usually about 1,500 deg. C. for breunnerite. It is sometimes assumed to be due to catalytic action, but this appears to be a misconception. However, an original content of ferrous carbonate does facilitate, by this change into magnesioferrite, conversion to artificial periclase, in part if not wholly, at a lower temperature than would otherwise be possible.

Microscopic examination of a well-sintered product from a mineral containing 4 per cent. to 5 per cent. of ferrous carbonate shows the existence of black particles of magnesioferrite as inclusions in the crystals of periclase, particularly towards their centres.

As magnesioferrite has a specific gravity between 4·57 and 4·65 its presence to the extent of several units per cent. gives a marked increase to the average gravity of the mass containing it. A test for specific gravity alone, unless considered in relation to chemical composition, is therefore not a safe guide to the extent of conversion to periclase, except in the case of a magnesite substantially free from impurities, especially iron.

So far the raw material for the refractory product has been assumed to be either relatively pure amorphous magnesite, which has been burned at a temperature of 1,800 deg. C. and upwards, or a ferruginous crystalline magnesite treated at about 1,500 deg. C. In practice there is very little dead-burning of amorphous magnesite without some form of preliminary adjustment to reduce its sintering temperature by providing conditions favourable to the formation of magnesioferrite. This usually involves an admixture of ferric oxide, which may be in the form of iron ore, roll scale, etc. If this addition is made to the raw magnesite, crushing is necessary so that incorporation may be as complete as possible. In such case a rotary kiln must be used for firing.

A recent patent provides for a first calcination to the caustic stage, mixing this product with a calculated proportion of ferric oxide, briquetting the mixture, and refiring to a sintering temperature of about 1,600 deg. C. in a shaft kiln. Another patented process covers an addition of limestone and ferric oxide, which causes first the formation of calcioferrite and then the later conversion of this to magnesioferrite. Magnetic iron ore is added to an unusually pure crystalline

magnesite at a production plant in the United States. In my view, however, there is too widespread a tendency to consider the typical sinter from Styrian breunnerite as the standard of quality. It is admitted that the steel industry has been greatly indebted to this material for a generation or more, but with modern advances in metallurgical practice, and especially the more general use of the electric furnace, there is an increasingly urgent call for the best refractories which are obtainable. The addition of a controlled amount of ferric oxide to the purer forms of magnesite does, to some extent, provide a better refractory material because the iron content is usually lower than in the product from breunnerite, but its chief justification lies in the lowering of working costs when amorphous magnesite is the raw material. The best refractory magnesite is undoubtedly that made from the purest mineral, with no factitious additions, treated at a temperature high enough to induce effective sintering and maximum contraction. This is possible in a shaft kiln, especially with oil firing, but the finest product is that which has been taken to its fusing point in the electric furnace. This high temperature treatment, whatever form it takes, will obviously entail an increase in working costs.

Bricks.

Magnesite bricks for the metallurgical industries were formerly made by the addition of an organic binding agent to the crushed sinter before moulding. This was destroyed in the refiring process. In modern practice the grain size of the crushed sinter is controlled and, after preliminary " souring " with water, the damp mass is moulded under powerful hydraulic presses. The green bricks so produced are strong enough to stand handling and stacking for refiring either in tunnel or chamber kilns.

When the raw material contains an appreciable amount of impurity, as is the case with breunnerite, the iron enters into combination as magnesioferrite, which appears as inclusions in the grains of periclase. A complex of silicates of calcium and magnesium and sometimes of aluminium, of lower melting point than the sintered magnesite, is formed and provides a glassy matrix, cementing the brick together. It follows, therefore, that the refractoriness of the brick is limited by the refractoriness of the bond. On the other hand if a pure magnesite is sintered at, say, 1,800 deg. C. and the bricks made from it are refired at a temperature between 1,700 deg. C. and 1,750 deg. C., the grains appear to cohere by inter-crystallisation and the refractoriness of the brick is practically that of the sintered magnesite from which it is made. The difference in the bonding process in the two types of brick is analogous to that between soldering and welding.

Le Chatelier and Bogitch made some comparative tests on magnesite bricks of (A) Styrian breunnerite containing MgO 86·7 per cent. and Fe_2O_3 6·0 per cent., and (B) Grecian amorphous magnesite containing MgO 93·4 per cent. and Fe_2O_3 0·5 per cent.

The failing loads per square inch were as follows. These results speak for themselves :—

Deg. C.	Styrian	Grecian
15	2,062 lb.	5,974 lb.
1,500	51 lb.	2,631 lb.
1,600	26 lb.	114 lb.

Conservation of Carbon Dioxide.

Substantially 50 per cent. of the weight of the mineral is evolved as CO_2 when decomposition takes place in the kiln, and it is rather surprising that more attention is not being given to the salving of this gas. Ordinarily the calcination of magnesite yields one ton of useful product from about 2·2 tons of raw material, and economic considerations therefore operate against treatment otherwise than near the source of supply. With the rapidly extending use of solid carbon dioxide as a refrigerant it is apparent that a profitable secondary commercial outlet should be attractive as the mineral could be transported for treatment in the vicinity of ready markets. This applies especially to a tropical country like India, where there are still very few organised facilities for refrigeration. Calcination, with recovery of CO_2, may be effected in two ways: (*a*) In closed containers or muffles, from which the gas is delivered in a high state of purity and needs very little after-treatment, but the unit yield is low. A 6-muffle kiln, with an input of about 1,000 tons a year, would only give about one ton a day of recoverable gas. (*b*) In shaft kilns, usually producer-gas fired, where the whole efflux of gases is tapped. The yield is high but contaminated with the gaseous products from the combustion of the fuel, and special purification plant, involving absorption into potash solution, is necessary. In addition to the CO_2 from the magnesite, there is also that from the furnace gases and a medium size kiln, with an output of about 500 tons of calcined product per month, should give a similar amount of recoverable CO_2.

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Ball-Mill Grinding.

An important contribution to the subject of grinding raw materials in the cement and other industries is contained in a paper entitled " Ball-Mill Grinding," by Messrs. W. H. Caghill and F. D. DeVaney (of the Metallurgical Division of the United States Bureau of Mines), issued as Technical Paper No. 581 by the United States Department of the Interior, Bureau of Mines, Washington, price 15 cents. The following is an abstract of the paper.

Samples.—Three " ores " were used in the experiments, and the size analyses are shown in *Table* 1. Chert was obtained as jig tailings from the Tri-State zinc district, and it is doubtful whether material more resistant to grinding can be found among the world's ores. The material had only 0·4 per cent. passing through a 200-mesh sieve. The dolomite was from the south-east Missouri lead district

TABLE 1.—Sizing Analyses of Feed Samples.

Size, mesh	Weight, percent cumulative			Size, mesh	Weight, percent cumulative		
	Chert	Dolomite A	Dolomite B		Chert	Dolomite A	Dolomite B
8	1.7	0.9	0.2	48	96.2	97.3	85.6
10	25.2	15.3	4.2	65	97.9	98.2	90.8
14	55.0	50.8	19.3	100	98.9	98.6	94.3
20	76.7	79.5	42.2	150	99.4	98.8	96.1
28	87.2	90.2	61.7	200	99.6	99.0	97.1
35	93.4	95.4	76.1	−200	.4	1.0	2.9

and was also well deslimed. Dolomite A had only 1 per cent. passing through a 200-mesh sieve ; also, like the chert, about 80 per cent. of its weight was on three consecutive sieves. The good sizing is due to the combined effect of the hydraulic classifier and to the concentrating table from which the sample was taken. The chert and dolomite A illustrate to what extent fine material can be sized without screens. The dolomite is about three times as grindable as the chert. Dolomite B is the laboratory drag " sand " accumulated from grinding tests on dolomite A. None of these materials had more than 1 per cent. of sulphides and hence they may be regarded as one-mineral products. The advantage in selecting two samples of such different grindability was not appreciated until the work was well under way. By the rotation practised, many fundamentals not discernible by testing a single ore were brought out.

Ball, Rod, and Pebble Mills.—Both cylindrical and conical mills were used. The cylindrical mills had the following dimensions : 19 in. by 36 in. unit type, 19 in. by 36 in. sectionalised with a grid, 24 in. by 24 in., and 24 in. by 36 in. Three conical mills were used. Two of these were made by lining a 24-in. by 36-in. drum. The first was lined so that the dimensions were 22.5 in. by 16.5 in. by 36 in. The second was lined so that it contained a succession of three truncated cones, all apexing toward the discharge end. Each of the

three truncated cones was 21 in. by 17 in. by 12 in., so that the total length was 36 in. The third conical mill had dimensions of 23·5 in. by 11·6 in. by 72 in. None of the mills was equipped with liners, as commonly conceived by operators; in fact, the shells were the liners, but lifters with moderate projections were always employed. Drum feeders were used in preference to scoop feeders.

The mounting of the mills was of the tyre-and-roller type, thus leaving the ends accessible for changes. The mills were driven by spur and pinion with the spur wheel attached to the head. The pinion was mounted on a countershaft with a silent chain drive connecting it to a direct-current motor. Roller bearings were used throughout. The motor losses and friction losses (dead load) of the 19-in. by 36-in. mill, after all possible means had been employed to reduce friction loss to a minimum, was considerable.

Ball Loads.—The ball loads are illustrated in *Table 2*. The first two loads have the designation "Davis" because Davis[1] developed the formula from which

TABLE 2.—BALL LOADS.

Size, inches	Weight, percent				
	Davis		Rationed		
	No. 1	No. 2	No. 1	No. 2	No. 3
2½	28		3	12	10
2	40	34	3	13	10
1½	18	36	6	14	10
1¼	7	15	9	15	11
1	7	8	15	21	16
¾		7	64	25	20
½					23
	100	100	100	100	100
Average weight, pounds	0.655	0.306	0.0632	0.153	0.086
Diameter of average ball, inches	1.658	1.285	.835	1.02	.854

the size analyses were derived. He took into account rate of wear of the different sizes and derived a load balanced against that wear when the renewal and rejection sizes were given. His formula considered nothing but "balance." He did not question the adaptability of his certain distribution of ball sizes to the particular distribution of particle sizes. In the present paper an attempt has been made to ascertain the amount of a given size of balls required for an estimated amount of a given particle size of ore, and this new load is called the "rationed load." The rationed loads are the result of a more or less scientific system of reasoning in which the ball size is not only best adapted to the particle size but also the amount of balls is rationed to the amount of the particle sizes and takes into account the finishing size. After the spherical balls, other grinding media were used. They were tetrahedrons, worn and battered balls, flint pebbles, sillimanite balls, rods, and pipes stuffed successively with materials of different density to give grinding media of different weights while the dimen-

[1] DAVIS, E. W., *Fine Crushing in Ball Mills: Trans. Am. Inst. Min. and Met. Eng.*, vol. 61, 1920, pp. 250-296.

sions remained the same. The grinding medium and the ore will be called "load" and "charge" respectively.

Screens and Flakiness.—Square-mesh screens were used to obtain the sized products in the various tables. At an early date, flakiness had been studied by screening on square-hole screens, then screening each size on a "rectang" screen of a selected size. The amount that passed the rectang gave a measure of flakiness. Flakiness varies in successive steps of grinding. This procedure and other methods for flakiness tests would be necessary if absolute surface values were required or if different types of grinding machines needed close scrutiny, but in this work it was deemed desirable to keep to the regular methods until more elementary things had been worked out. The work has been conducted in such manner that, although many surface calculations are made, they are little more than supplementary. That is, the ultimate results as based on square-hole screens may be seen at a glance, then surface calculations are made so as to combine the various characteristics into one numerical value. The necessity of subsieve examinations has been minimised by conducting the comparative tests so that each product of a series has about the same amount passing through a 200-mesh sieve. It is fully realised that products from grinding a given ore by diversified methods might have exactly the same amount of the minus 200-mesh size and yet have a different distribution of the subsieve sizes. In fact, the evidence is that the distribution would be different because (when the amount of minus 200-mesh material is the same) the distribution of the sieve sizes is different, hence the distribution of the subsieve sizes would be expected to be different.

Units of Surface.—The grinding rate or capacity and the grinding efficiency are expressed as surface tons per hour and surface tons per horsepower-hour respectively. This method has been used for several years by one of the largest mining companies. The method simply applies the well-established inverse ratios, that is, for example, the amount of surface in a unit weight of a ½-in. product is twice that of a 1-in. product and, similarly, the amount of surface in a unit weight of a 200-mesh product is twice that of a 100-mesh product. It was convenient to set the number 1 as a unit of surface of a unit weight of 1-in. material. By inverse ratio 361 is the units of surface in a unit weight of 150-mesh to 200-mesh product. These units may be extended beyond the subsieve sizes after elutriation. Absolute values may be assigned after elutriation if they are available, but for comparisons the relative values given are just as good.

The uncertainties of assigning units of surface to the subsieve sizes which have not been elutriated has caused some investigators much anxiety. Sometimes the assignments have been guarded by the term "useful grinding," which meant that the machine was given the same credit as if the subsieve particles were only small enough to pass the last sieve. Reporting in terms of useful grinding is still practised in this paper. Obviously this method fails to give the machine its full due. In this report tests are compared only when the amounts of subsieve sizes are nearly the same. Many opportunities have been given to learn what

is meant by "amounts of subsieve sizes are nearly the same." Repeat tests had to be made to get this sameness in the minus 200-mesh products. If tests were of the batch type the correct time period had to be found by trial, or if they were of the continuous type the proper feed rate had to be found. Complete calculations were always made, whether or not the results were to be used for final record. The conclusion was reached that if the percentages of minus 200-mesh products did not differ by more than 3 per cent. the test could be recorded for final comparison.

At first thought it would seem that if two tests, one with 10 per cent. and the other with 13 per cent. minus 200-mesh material, are to be compared by means of the surface calculations for useful grinding the one with 10 per cent. minus 200-mesh material would yield the higher efficiency values. But this is not necessarily true; the test with 13 per cent. might show the higher efficiency because the grinding medium was too small and had to work inefficiently in the first part of the period and only began to work with advantage after some reduction of particle size had taken place. An attempt to assign absolute values, as, for example, kg.-cm. per cm.^{2} to the calculated values obtained in this paper would not only be useless but it would lead to conclusions that are basically wrong. Only when thorough elutriation, or its equivalent, of the subsieve sizes has been practised can absolute surface values be assigned. Now that the general principles of useful grinding have been gleaned, the future work of seeking fundamentals will be studied by applying surface values obtained by extending the relative values into the subsieve sizes. When that is done the densities of the various minerals will enter into the calculations.

In this paper, chert and dolomite have been treated as if they had the same density, when in reality a unit weight of a given screen size of chert would be expected to have more surface than dolomite.

Methods for calculating useful grinding will always be serviceable but they are not wholly comprehensive, and with the passage of time more and more need for dissection of subsieve sizes will be felt. The size analyses in the tables are cumulative, this step having been performed after the surface calculations were completed.

Power.—Power is one of the most enlightening single factors in the study of grinding. In plant operations gross power input to the ball-mill unit is the quantity of economic interest to the operator, but from the scientific aspect the study must be made of the net power expended within the mill. The difference in these two values is motor inefficiency and dead (friction) load. Repeated dead-load tests have been made in the Rolla laboratory, and a similar test was made in a concentrator. This was done by packing the working load centrally in the mill so that it revolved like a flywheel. In the laboratory two 220-volt direct-current motors were used interchangeably. One was 3-h.p. shunt-wound and the other was 5-h.p. compound-wound. All powers recorded in this paper are net power unless otherwise stated.

Critical Speed and the Power-speed Curves.—Critical speed is the speed at which the centrifugal and centripetal forces, acting on an infinitely small particle travelling on and with the breast of the mill, are equal. Then the particle will not leave its position. It becomes part of a " flywheel." The phenomenon is independent of specific gravity. Obviously, on account of their greater axis of revolution, small particles of ore will centrifugalise at lower speeds than large balls. Balls never centrifugalise at speeds indicated by the formula

$$\text{C.S.} = \frac{54{\cdot}19}{\sqrt{r}},$$

where r is the radius of the mill in feet, because, first, they are not infinitely small and, second, they slip and may never attain the speed of the breast of the mill. Hence the term " theoretical critical speed " is best when a safeguard in expression is required. In spite of the lack of a close analogy between the deportment and the formula, critical speed is a valuable anchor in comparing mills of different diameters. In this paper the term will be designated as " per cent. critical," or " per cent. speed."

In power-speed curves the percentage speed that is plotted is the speed of the mill in accord with the formula, although the speed of the balls may lag far behind. Tumbling tests with a wire screen on the end of the mill will indicate the lag. The investigator need not worry about undue lifting of the balls by the screen ; if he selects a mill 2 ft. long with a sheet-iron head on one end and a screen on the other, and uses a flashlight to view the interior, he will see that the lifting at each end is a little higher than at the middle. That is, the charge sags at the midway position, but the sagging is not enough to make examination confusing. On account of the lifting effects of the ends, a mill 2 ft. long is expected to take a little more than half as much power as a mill 4 ft. long.

Too much emphasis cannot be laid on the fact that the peak of a power curve comes at a lower speed than that which centrifugalises the outer circle of balls. The power peak comes when the outer circle of balls strikes the breast of the mill in what may be called the 8 o'clock position. The power curve drops with regularity when the impingement is higher or lower. An extra amount of keying would cause a higher impingement but, on the other hand, slipping would reduce the elevation. Because of moderate action, power is below maximum when the impingement is below the 8 o'clock position, and because of excessive action, which results in the giving back of much of the energy, power is again below maximum when the impinging is above the 8 o'clock position. There is nothing in the power-speed curve of a big mill to indicate the speed at which centrifugalising begins, but squirrel-cage tests have shown that maximum power is consumed at a much lower speed than is necessary to centrifugalise the balls.

Selective and Nonselective Grinding.—The terms " impact " and " attrition " are not satisfactory for designating types of grind. The reason will be obvious when it is seen that high speed and low speed gave about the same type of grind. Furthermore, the term attrition is not as specific as formerly when it was used to signify the undesirable work of excessively small media ;

in batch tests, when the amounts of subsieve material were the same, the excessively big media left too many coarse particles of ore and in that respect failed as would excessively small balls. Attrition cannot apply to the failure of the large balls. " Nonselective " is a better term because it covers both extremes of poor work, and " selective " is descriptive of good work on the coarse material.

Selective and nonselective grinding are used to compare products that have the same amount of the subsieve size. Then the product with the least amount of coarse sizes shows good selective grinding and the others are ground nonselectively. Stage grinding, by repeated passes followed by removal of the finished material, is the best means of obtaining selective grinding. These

TABLE 3.—RELATION OF POWER TO SPECIFIC GRAVITY OF GRINDING MEDIA (STUFFED PIPES) AND TO THE WEIGHT OF TOTAL CHARGE AT THE RESPECTIVE SPEEDS.

(Mill, cylindrical, 19 by 36 inches. Circuit, continuous, open. Discharge, 8 inches. Speed, varied. Load, pipes, 1⅜- by 35-inch. Weight, varied. Volume, 42 per cent. Ore charge, chert. Consistency, 60 per cent. solids.)

Critical speed, percent	Horsepower (A)	Grinding media specific gravity (B)	Total charge of pipes, ore, and water, pounds (C)[1]	Ratio B/A	Ratio C/A
40	0.422	2.89	433	6.8	1026
	.549	4.17	585	7.6	1066
	.667	5.00	684	6.5	1025
	.863	6.66	881	7.7	1021
	.900	7.30	960	8.1	1067
50	.540	2.89	433	5.3	802
	.721	4.17	585	5.8	811
	.874	5.00	684	5.7	783
	1.110	6.66	881	6.0	794
	1.160	7.30	960	6.3	828
60	.640	2.89	433	4.5	677
	.883	4.17	585	4.7	663
	1.050	5.00	684	4.8	651
	1.300	6.66	881	5.1	678
	1.400	7.30	960	5.2	686
70	.780	2.89	433	3.7	555
	1.071	4.17	585	3.9	546
	1.250	5.00	684	4.0	547
	1.540	6.66	881	4.3	572
	1.690	7.30	960	4.3	568

[1] Weight of ore and water was 90 pounds.

terms must not be confused with differential grinding, which has to do with the relative grinding rates of two or more minerals in an ore.

Relation of Density of Grinding Medium to Power and Type of Grind. —Earlier work on the use of pipes (instead of rods) stuffed with different materials to give grinding media of different densities when the dimensions were always the same showed that through a range of densities from 2·89 to 7·30 and a range of speeds from 40 to 70 per cent. critical, the size analyses of the products were about the same when the ratio of tons of feed to horsepower was constant. These results seemed to be a step toward the answer to an old question about the effect of the density of the grinding medium. These considerations are carried further by reference to *Table* 3, which has additional data on the same tests.

The tests were made at four speeds, and at each speed the pipes had five different loadings to give the specific gravities indicated. The power is recorded under A and the densities of the stuffed pipes are under B. The column $\frac{B}{A}$ is notable because of the moderately regular variation of the values, but when taken by itself does not permit a satisfactory interpretation. However, when the weight of ore and water is taken into account in C, and the ratio $\frac{C}{A}$ is given as in the last column, ratios of remarkable accord are obtained. From these ratios it is concluded that under the conditions specified the power varied as the weight of medium plus ore plus water. Doubtless the weight of the ore in a mill is a factor in power consumption.

TABLE 4.—ROD-MILL GRINDING WITH STUFFED PIPES OF DIFFERENT DENSITIES.

(Mill, cylindrical, 19 by 36 inches. Circuit, continuous, open. Discharge, 8 inches. Speed, 50 per cent. critical. Load, pipes, 1⅜- by 35 inches. Weight, varied. Volume, 42 per cent. Ore charge, chert. Consistency, 60 per cent. solids.)

Size, mesh	Feed, weight percent	Weight, percent				
		Specific gravity of grinding media				
		2.89	4.17	5.00	6.66	7.30
10	25.2	2.5	2.1	1.7	1.2	0.9
14	55.0	16.0	16.1	14.2	13.2	11.2
20	76.7	37.7	38.4	37.2	36.4	35.8
28	87.2	53.2	54.2	53.6	52.8	53.2
35	93.4	66.0	66.7	67.0	66.2	67.2
48	96.2	73.6	74.2	74.9	74.1	75.1
65	97.9	79.7	80.0	81.0	80.3	81.6
100	98.9	84.2	84.4	85.5	84.9	86.2
150	99.4	87.5	87.7	88.8	88.3	89.5
200	99.6	89.5	89.7	90.8	90.5	91.5
−200	.4	10.5	10.3	9.2	9.5	8.5
Surface, tons per horsepower-hour		15.6	15.6	14.7	15.8	15.2
Ton per hour		.092	.128	.155	.207	.220
Horsepower		.477	.650	.790	1.02	1.06
Ton per horsepower-hour		.193	.196	.196	.203	208

The difference in size analyses mentioned are shown in *Table* 4, which is intended to show how much the light media failed. The results of grinding at 50 per cent. critical are brought together. The selection of any other speed would serve the purpose as well. Although the calculated efficiencies are about the same at the various densities of media, it is plain that the light media gave nonselective grinding; that is, the products from the light pipes had too much in the coarse sizes. This failure is not brought out by the surface calculations because the change in amount of calculated surface is so small when coarse material is crushed to an intermediate size. Nonselective grinding by the light media inspires the idea that whereas much attention of operators has been given to the size of media for certain particle sizes of ore, the weight of the media also should be taken into account. That a light medium fails by nonselective grinding is shown later in *Table* 21, in which pebbles are considered. There the pebbles failed when the ore was very hard.

(*To be continued.*)

Refractories for Cement Kilns.*

By HENRY G. FISK, Ph.D.

(72) K. Endell. Dilatation and sensitivity to heat of refractory brick in the cement industry. *Rev. mat. constr. trav. pub.*, No. 270, pp. 111–15 (1932). *Cer. Abs. 11*, No. 7, 415 (1932).—Besides the continuous variation in length of refractories with changes of temperature, there is another kind of dilatation which depends on a reversible change in the physical state of some crystalline constituents (a change connected with a variation in volume). The latter kind of dilatation is especially important in the SiO_2 system. Tables show changes in specific gravity, specific volume, increase in volume and length of α-quartz, α-cristobalite, α-tridymite, β-cristobalite, and quartz glass; also a linear dilatation conditioned by the transformation of different forms of SiO_2 into each other at high temperatures. Besides this variable dilatation and permanent dilatation a permanent contraction exists which is determined by the shrinkage of clay particles fired at insufficiently high temperatures. The importance of these tensions depends on the size and volume of the brick, the co-efficient of dilatation, and variations of temperature. When tensions exceed the limit of resistance they produce disintegration or even destruction of the structure of the brick. The property of the brick to react against a change of shape or volume due to pressure, tensile strength, or torsion, produced by irregular heating or cooling, is elasticity. When the limit of elasticity is exceeded a permanent variation in length appears (plastic deformation). The limit of elasticity is highly important in the case of a growing sensitivity of refractory bricks to heat. If, during the increase of temperature, the limit of elasticity is exceeded and a permanent variation in state occurs, then the tensions within the brick equalise and the brick is no longer as sensitive to temperature. All refractories are more stable at temperatures higher than 650 deg. C; this fact was substantiated in practice where it was observed that all refractories are unaffected by temperatures above dark red. Test to ascertain the resistance of refractory products to temperature variations are described.

(73) A. E. Fitzgerald. Unfired magnesite refractories in rotary cement kilns. *Pit and Quarry, 24* (2), 38 (1932). *Cer. Abs. 11*, No. 7, 417 (1932).—Highly-aluminous bricks are superior to clay bricks because they suffer less from vitrification and form more refractory mixtures with cement clinker. Mixtures of cement clinker and unfired magnesite brick are even more refractory than clinker-high-alumina-brick mixtures, and so allow the operator to fire at much higher temperatures than heretofore.

(74) P. Leone. Silico-aluminous materials for lime kilns. *Ann. chim. applicata, 22*, 397–404 (1932). *Cer. Abs. 12*, No. 3, 109 (1933).—Poor resistance of CaO-kiln refractories is considered in relation to the ternary system, CaO-Al_2O_3-SiO_2 [cf. *Brit. Chem. Abs., 11*, 50, (1915)]. Proportions of Al_2O_3 : SiO_2 which lead readily to a low-melting eutectic with CaO should be avoided. The refractory should be compact and uniform, without cracks, etc., which facilitate attack by CaO.

(75) W. N. Jung. Sintering of cement-clinker brick. *Zement 21* (30), 433–38

* Concluded from December, 1939, January and March, 1940.

(1932). The character of the changes occurring in cement-clinker brick for the lining of the sintering zone of revolving furnaces is described and the reasons for the low resistance are determined. Experiments for changing these bricks into a sintered clinker mass to obtain a resistant lining are discussed. The testing of the resistance to changes of temperature of vitrified clinker bricks (under conditions similar to those occurring in revolving furnaces) showed that they are from 9 to 14 times more resistant than ordinary unfired cement-clinker bricks.

(76) D.S. Refractory lining for rotary cement furnaces. *Corriere ceram., 13* (11), 411—13 (1932). *Cer. Abs. 12*, No. 4, 160 (1933).

(77) W. Eitel. Thermotechnological principles of cement kilns. *Ber. deut. keram. Ges., 13* (3), 150 (1932). *Cer. Abs. 11*, No. 9, 480 (1932).

(78) K. Endell. Use of magnesite units resistant to thermal shock for the sinter zone of cement, shaft and rotary. *Zement 21*, 522 (1932). *C.A. 27*, No. 1, 175 (1933).—Suggests that a new magnesite brick which is described in Stahl u. Eisen *52*, 759–63, and which is resistant to temperature changes, should prove economical in the cement industry.

1933

(79) J. Fauner. Refractory Material for Cement Rotary Kilns. *Cement and Cement Manuf. 6*, 113–23 (1933). *Rock Prod. 36*, No. 6, 42 (1933); *C.A. 27*, No. 19, 4898 (1933). A general discussion of thermal and chemical effects on kiln linings. Causes of destruction of kiln linings are (1) mechanical effects, (2) thermal changes, (3) chemical reactions at high temperatures. The hardness of the block is important to resist the abrasion of the clinker and the raw mix. The importance of thermal expansion is discussed. The Corhart Electrocast in the hot zone has 4 to 6 times the life of ordinary brick.

(80) Anon. Lining cement kilns for manufacturing high-quality cements. *Tonind. Ztg., 57* (7), 75–76 (1933). *Cer. Abs. 12*, No. 5, 194 (1933).—High-temperature as well as low-temperature clinkers require an expensive furnace lining of high refractoriness, good mechanical resistance, and low porosity. No satisfactory lining is available for clinkers obtained by the addition of fluxes to ordinary raw materials.

(81) P. M. Tyler. Magnesite and its compounds in 1931. *Mineral Resources of the U.S.*—U.S. Bureau of Mines, Washington. Part II, 263–77 (1933).—

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(82) Anon. Magnesite lining. *Rock Prod.* 37 (1), 85 (1934).—Ritex in the hot zone of a Mexican cement kiln gave 2·4 times longer life than other linings without failure. In New England a 15 ft. hot zone lining gave 310 days compared with a previous average of 54 days. Ritex is unburned hydraulically pressed magnesite.

(83) P. P. Budnikov and S. A. Shicharevich. The selection of a lining for rotary kilns used in the preparation of Portland cement and sulphuric acid from gypsum and clay. *Tonind.-Ztg. 58*, 71–2, 83–6 (1934). *C.A. 28*, 3551 (1934).—Chromite-magnesite bricks and black dinas are recommended.

(84) R. P. Heuer. Petrographic, fusion, and slag studies of magnesite refractories in rotary kiln service. *Rock Products 37* (12), 42–46 (1934).—Unburned magnesite brick in rotary kilns burning Portland cement and similar basic materials is preferable to that of 70 per cent. alumina brick. These bricks show fusion temperatures in the presence of Portland cement and dolomite clinker approximately 900 deg. higher than 70 per cent. alumina bricks.

(85) W. Czernin. Hydraulically hardening refractory materials. *Deutsch. Keram. Ges. Ber. 15* (9), 463–71 (1934).—Various methods of obtaining monolithic wall surfaces in kiln construction are reviewed.

1935

(86) J. C. Crawford, Jr. How rotary cement kilns are insulated. *Concrete Mill S. 43* (10), 35–8 (1935).—Describes insulation in Portland cement kiln exclusive of hot zone. Reports fuel saving and increased production.

(87) J. Dautrebande. Lining for rotary kilns. *Rev. Mat. Constr. 305*, 37–41 (1935).—Describes the manufacture and installation of cement-clinker blocks in a cement rotary kiln.

1936

(88) D. Steiner. Refractory lining for cement kilns. *Zement 25* (37), 619–24 (1936).—A discussion of the properties desirable in rotary and shaft kiln linings leads to an account of the composition and behaviour of various proprietary and other refractory products.

1937

(89) I. E. Gurvich. The increase of the stability of clinker-concrete lining of rotary cement kilns. *Tsement 5* (4), 67–70 (1937). *Chem. Abs. 32* (5), 1893 (1938).—For Portland cement clinker, a 5 per cent. addition of tripoli is not sufficient to bind all free lime; with a 10 per cent. addition the spalling resistance remains unchanged after three months' air storage, and a still higher resistance is attained with a 25 per cent. addition.

1938

(90) W. F. Rochow. Magnesite linings in kilns. *Rock Prod. 41* (4), 82–83 (1938).—Describes successful installations of chemically-bonded magnesite brick in cement and dolomite kilns.

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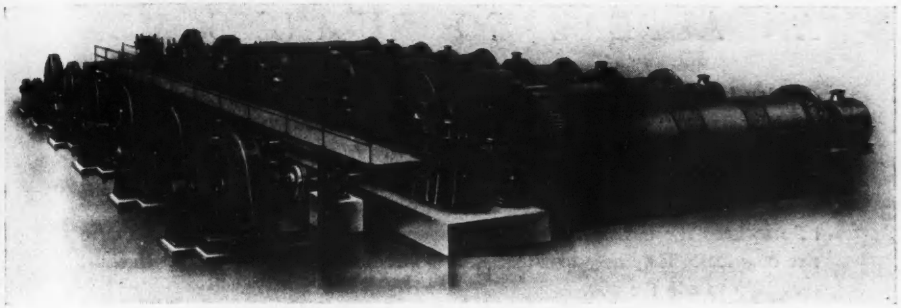

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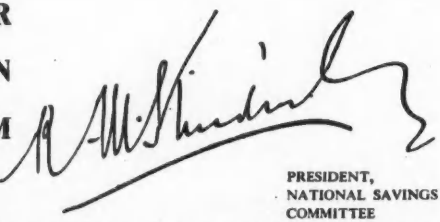

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